

CLAIMS

1. Method of producing phosphoric acid, comprising

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- at least one phosphate ore attack with a first aqueous solution of hydrochloric acid, with the formation of an attack liquor,

10 - a first separation, in the attack liquor, between an insoluble solid phase containing impurities and a separate aqueous phase comprising in solution phosphate ions, chloride ions and calcium ions,

15 - an extraction of an aqueous solution containing phosphate ions, chloride ions and calcium ions by an organic extraction agent, in order to form an aqueous extraction phase comprising chlorine ions and calcium ions and an organic extraction phase containing phosphoric acid, and

20 - a re-extraction of the organic extraction phase by an aqueous re-extraction agent, in order to isolate an aqueous re-extraction phase containing phosphate ions, as well as, possibly,

- a concentration of the aqueous re-extraction phase in order to form an aqueous solution of pure phosphoric acid,

25 characterised in that it also comprises

- a neutralisation of the said aqueous phase separated from the attack liquor by the addition of a calcium compound in order to form with the said phosphate ions a calcium phosphate insoluble in water, which precipitates,

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- a second separation, in the said neutralised aqueous phase, between an aqueous phase comprising in solution calcium ions and chloride ions and a precipitated solid phase based on the said calcium phosphate insoluble in water, and

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- a solubilisation of at least part of the said precipitated solid phase separated, in a second aqueous solution of hydrochloric acid, with the formation in the said aqueous solution containing phosphate ions, chloride ions and calcium ions to be extracted by means of an organic extraction agent.

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2. Method according to Claim 1, characterised in that the said first aqueous solution of hydrochloric acid has an HCl concentration of no more than 10% by weight, advantageously 3% to 6%, preferably 3% to 5%.

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3. Method according to one of Claims 1 and 2, characterised in that, in the attack liquor, the molar ratio between HCl and Ca is between 0.6 and 1.3.

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4. Method according to one of Claims 1 to 3, characterised in that the attack step is performed at ambient temperature.

5. Method according to any one of Claims 1 to 4, characterised in that the calcium compound of the neutralisation step is chosen from amongst the group consisting of calcium hydroxide, calcium oxide and water-soluble calcium salts, such as calcium carbonate, and in that the calcium phosphate insoluble in water is calcium monohydrogenophosphate (DCP).

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6. Method according to any one of Claims 1 to 5, characterised in that the said separated precipitated solid phase, based on the said insoluble calcium phosphate, has a concentration of 40% to 50% by weight P₂O₅ and 25% to 28% Ca.

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7. Method according to any one of Claims 1 to 5, characterised in that the said second aqueous solution of hydrochloric acid has an HCl concentration greater than that of the said first aqueous solution of hydrochloric acid, preferably 15% and at a maximum 20% by weight.

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8. Method according to any one of Claims 1 to 7, characterised in that the said solubilised aqueous solution, to be extracted, has a P₂O₅ concentration of 8% to 15% by weight, preferably 10% to 13%.

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9. Method according to any one of Claims 1 to 8, characterised in that it also comprises, after the said extraction, washing of the organic phase extracted by a fraction of the aqueous re-extraction phase, in order to eliminate from the organic phase extracted a hydrochloric acid and calcium chloride content entrained by it and any impurities still present.

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10. Method according to any one of Claims 1 to 9, characterised in that it also comprises a steam entrainment of traces of organic extraction agent from the aqueous extraction phase containing chlorine ions and calcium ions.

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11. Method according to either one of Claims 9 and 10, characterised in that the aqueous re-extraction phase containing phosphate ions has a P₂O₅ concentration of 15% and 25% by weight.

12. Method according to any one of Claims 1 to 11, characterised in that the phosphate ore has a coarse grain size, preferably substantially between 150 and 500 μm , and a P_2O_5 content of 15% to 38% by weight.

5 13. Method according to any one of Claims 1 to 12, characterised in that it also comprises a treatment of the said aqueous phase issuing from the second separation, containing in solution calcium ions and chloride ions, by means of an aqueous solution of sulphuric acid with the formation of insoluble calcium sulphate, which precipitates, and an aqueous phase
10 based on hydrochloric acid, an isolation of the calcium sulphate precipitate and an at least partial recycling of the aqueous phase based on hydrochloric acid in order to form the said first and/or second aqueous solution of hydrochloric acid.

15 14. Method of preparing a phosphoric acid salt, comprising

- at least one attack on phosphate ore by an aqueous solution of hydrochloric acid, with the formation of an attack liquor,

20 - a first separation, in the attack liquor, between an insoluble solid phase and an aqueous phase,

- a neutralisation of the said separated aqueous phase by the addition of a calcium compound in order to form, with phosphate ions contained in this
25 aqueous phase, a calcium phosphate insoluble in water, which precipitates, and

- a second separation, in the said neutralised aqueous phase, between a liquid phase and a precipitated solid phase based on the said calcium
30 phosphate insoluble in water,

characterised in that the attack on the phosphate ore comprises a dissolving of the phosphate in the ore, the attack liquor containing this phosphate in the form of phosphate ions, and in that the solid phase separated from the attack liquor contains impurities and the aqueous phase separated from the attack liquor contains the said phosphate ions thereof, chloride ions and calcium ions, this aqueous phase being subjected to the said neutralisation and second-separation steps.

15. Method according to Claim 14, characterised in that the said aqueous solution of hydrochloric acid has an HCl concentration of at most 10% by weight, advantageously 3% to 6%, preferably 3% to 5%.

16. Method according to one or other of Claims 14 and 15, characterised in that, in the said attack liquor, the molar ratio between HCl and Ca is between 0.6 and 0.13.

17. Method according to one or other of Claims 14 to 16, characterised in that the attack step is performed at ambient temperature.

18. Method according to any one of Claims 14 to 17, characterised in that the calcium compound of the neutralisation step is chosen from amongst a group consisting of calcium hydroxide, calcium oxide and water-soluble calcium salts, such as calcium carbonate, and in that the calcium phosphate insoluble in water is calcium monohydrogenophosphate (DCP).

19. Method according to any one of Claims 14 to 18, characterised in that the said separated precipitated solid phase, based on the said calcium phosphate insoluble in water, has a concentration of 40% to 50% by weight P₂O₅ and 25% to 28% Ca.

20. Method according to any one of Claims 14 to 19, characterised in that it also comprises a treatment of the said liquid phase issuing from the second separation and containing calcium ions and chloride ions in solution, by means of an aqueous solution of sulphuric acid with the
5 formation of insoluble calcium sulphate, which precipitates, and an aqueous phase based on hydrochloric acid, an isolation of the calcium sulphate precipitate and an at least partial recycling of the aqueous phase based on hydrochloric acid in order to form the said aqueous solution of hydrochloric acid.

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21. Method of producing phosphoric acid, comprising

- a solubilisation of a solid phase based on a calcium phosphate insoluble in water in an aqueous solution of hydrochloric acid, with the formation of a
15 solubilised aqueous solution containing phosphate ions, chloride ions and calcium ions,

- an extraction of the aqueous solution solubilised by an organic extraction agent, in order to form an aqueous extraction phase containing chloride
20 ions and calcium ions and an organic extraction phase containing phosphoric acid, and

- a re-extraction of the organic extraction phase by means of an aqueous re-extraction agent, in order to isolate an aqueous re-extraction phase
25 containing phosphate ions, as well as, possibly,

- a concentration of the re-extraction aqueous phase in order to form an aqueous solution of pure phosphoric acid.

22. Method according to Claim 21, characterised in that the aqueous solution of hydrochloric acid has an HCl concentration of from 15% to a maximum of 20% by weight.

5 23. Method according to one of Claims 21 and 22, characterised in that the said solubilised aqueous solution to be extracted has a P₂O₅ concentration of 8% to 15% by weight, preferably 10% to 13%.

10 24. Method according to one of Claims 21 to 23, characterised in that it also comprises, after the said extraction, a washing of the extraction organic phase by a fraction of the re-extraction aqueous phase, in order to eliminate from the extraction organic phase a hydrochloric acid and calcium chloride content entrained by it and any impurities still present.

15 25. Method according to Claim 24, characterised in that the re-extraction aqueous phase has a P₂O₅ concentration of 15% to 25% by weight.

20 26. Method according to any one of Claims 21 to 25, characterised in that the solid phase based on calcium phosphate insoluble in water is obtained by a method according to any one of Claims 14 to 20.